

Dem-Val of Electrodeposited Nano Co-P Alloys for NLOS Coating Applications at NADEP Jacksonville

Integran Technologies Inc.

HCAT Meeting, San Diego - January 25th, 2006

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Report Documentation Page

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Overview



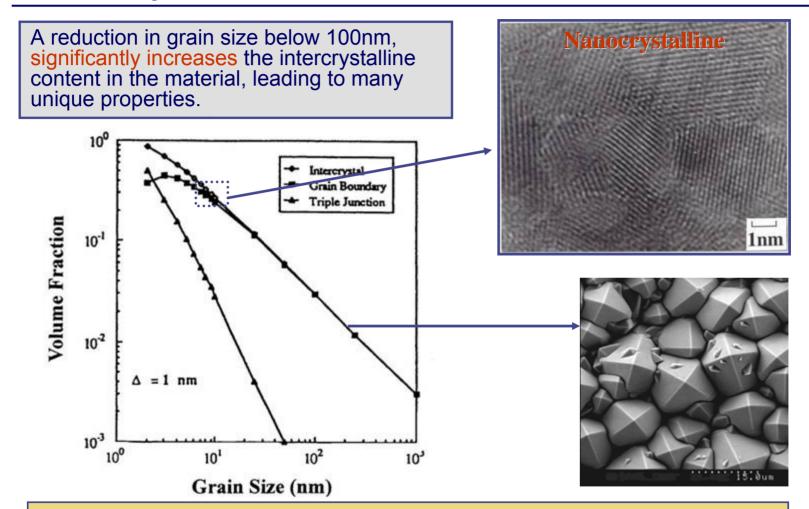
- Nanocrystalline Materials
- History of the nCoP NLOS Cr-replacement Project
- Overview of nCoP Process and Properties
- Scale-up of Tank Process
 - Installation at Integran
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- Summary

Technology Partnerships Canada

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Understanding the relationship between **Microstructure** and **Processing conditions** is **Critical**



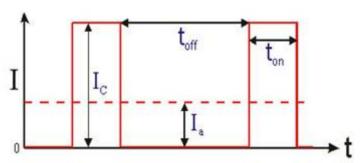


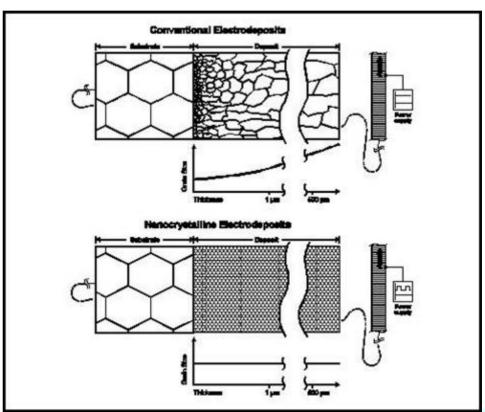
Electrodeposition of Nanomaterials

Electrodeposition provides a cost-effective synthesis method to produce high quality nanocrystalline materials.

Microstructure control achieved through modifications to bath chemistry (additives etc) and/or electrical parameters (pulse plating)

Pulse Plating favors
nucleation of new grains over
growth of existing grains,
resulting in an ultra-fine grain
structure throughout the
entire thickness of the
coating, right from the
substrate interface.







History of Cr-Replacement Project

Strategic Environmental Research and Development Program (SERDP) Project #PP-1152

<u>"Electroformed Nanocrystalline Coatings – An Advanced Alternative to Hard Chrome Electroplating"</u>

Technical Objective

Develop an environmentally benign advanced nanocrystalline based coating technology that:

- Is compatible with conventional electroplating infrastructure
- Will produce coatings that meet or exceed the overall performance of hard chrome (hardness, wear, corrosion, fatigue)
- Has costs similar to or less than life-cycle cost of existing hard chrome electroplating processes
- Can be applied to non-line-of-sight surfaces

Nano Co-P as EHC alternative was developed and demonstrated at the lab scale

Cobalt alloy selection

- Mechanical properties, High plating efficiency
- No constituents on EPA or AFMC lists of hazardous materials
- Longer term view





History of Cr-Replacement Project

Environmental Security Technology Certification Program (ESTCP) Project #PP-0411

"Nanocrystalline Cobalt Alloy Plating for Replacement of Hard Chrome and Thin Dense Chrome (TDC) on Internal Surfaces"

Technical Objectives

- Scale up to industrial production & move to depot
 - Demonstration sites: Naval Air Depot Jacksonville (NADEP-JAX) and Integran Technologies
- Develop nano Co-P selective plating as repair
 - Demonstration sites: Air Force Ogden Air Logistics Center (ALC) and Integran Technologies
- Develop nano Co-P based TDC alternative
 - Modify standard nCoP deposition process for ultra thin coating



Technology Description



Process

Simply an electrodeposition process

 Parameters modified to yield deposits with average grain sizes below 100nm using pulse plating

Plating Efficiency >90%

- High Deposition rates (2-8 mils/hr)
- 10x the plating rate of EHC at same power current density
- 1/10th the power consumption at the same plating rate

Consumable & nonconsumable anode

Cobalt Pieces in Ti basket, Graphite

Phosphorus Content: 0 to 10wt%

Controlled by solution chemistry and plating conditions







Solution Control and Maintenance

Similar to that required for nickel-plating solutions:

- Filtration
 - Control particles
- pH monitoring
 - Control deposit uniformity
- Surface tension control
- Solution density monitoring

Periodic maintenance procedures

- Activated carbon treatment
 - Organics
- Dummying (low current density plating)
 - Remove various metallic impurities







		Nanocrystalline Co-P	Hard Chrome
Hardness	As-Deposited	600-700 VHN	800-1200 VHN
	HT @ 250°C	700-800 VHN	-
			-
Ductility		2 – 7 % Elongation	<.1%
Thermal Stability		400°C	400°C
Wear	Adhesive (Pin-on-disk)	5-6 x 10 ⁻⁶ mm ³ /Nm (Alumina Ball on Nano Co-P Disk)	9-11 x 10 ⁻⁶ mm ³ /Nm (Alumina Ball on Cr Disk)
	Coefficient of Friction	0.5	0.7
	Abrasive (Taber)	18 mg / 1000 cycles (CS-17) 11 mg / 1000 cycles (CS-10)	3.2 mg / 1000 cycles (CS-17) 1.0 mg / 1000 cycles (CS-10)
Corrosion	Salt Spray	Protection Rating 7 @ 1000 hrs	Protection Rating 2 @ 1000 hrs







Scale up of Tank Process



Industrial Scale Up at Integran Technologies

NTEGRAN

Tank

Dimensions (3'x5'x3') (~1300L)

- 1" Polypropylene
- Separate well to house pump and filter
- 3/4hp pump
- 1µm filter
- 60kw heaters (Ti) digitally controlled

Power supply

- Integran's specifications
- Peak current 1500A
- Average current 500A
- Pulse timing (ton and toff)
 - 0-100ms, Δt=0.1ms
- Verified at Integran and shipped to NADEP-JAX







Industrial Scale Up at Integran Technologies

Dem/Val Studies

- Integran 1300 L Bath has been in operation for 14 months, >17,000 Amp-hrs, No major problems
- Process Verification
 - Scaled-up process produces acceptable nanostructured coatings on small and large test coupons
 - Verified on large test pieces (4" ID, 1' Length)
- Operating Window Identification
 - Optimal Bath chemistry identified
 - Optimal Pulse Conditions identified
 - Temperature, Current density and pH operating ranges identified
- Contaminant Study
 - Completed a lab scale study of the effects of various metallic contaminants in the plating solution (Cr⁶⁺, Cr³⁺, Fe²⁺, Fe³⁺, Cu²⁺, Ca²⁺, Ni²⁺, Na⁺, F⁻, and SO₄²⁻)
- Operating Manual has been Prepared for Transfer to NADEP-JAX

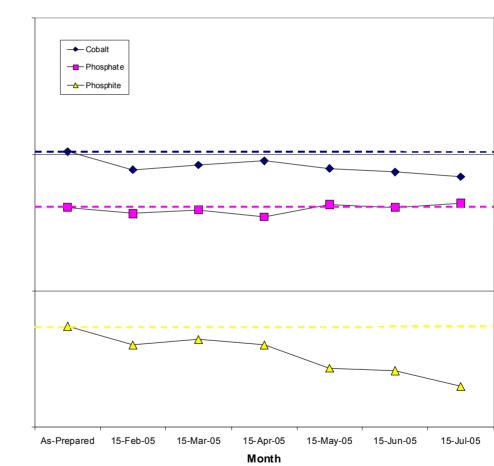


nCoP Bath Maintenance and Monitoring

Log Concentration (mol/L)

Chemical analysis of plating solution

- A sample of the 1300L bath is sent for chemical analysis on a monthly basis
- Key bath ingredients are monitored by ICP/IC
- Results to date show cobalt and phosphate are relatively stable while the phosphite ions decrease with use of the bath.
- This is expected due to the consumption of P







nCoP Bath Maintenance and Monitoring

Chemical analysis of impurities in the plating solution

- Impurities in the plating solution are analyzed on a monthly basis by ICP/IC
- Impurities showing the highest levels include: Al, Fe, Li, Na and Ni
- Copper bus bars and Titanium heaters are not negatively affecting the bath chemistry

Impurities > 1 ppm	15-Feb-05	15-Mar-05	15-Apr-05	15-May-05	15-Jun-05	15-Jul-05
Aluminum	2.40	3.10	4.58	3.01	6.73	9.14
Arsenic	18.10	-	7.70	3.11	-	<0.01
Barium	1.26	1.23	1.39	1.33	1.37	1.29
Calcium	-	-	-	4.20	5.63	<1.00
Copper	6.71	7.40	3.99	3.78	2.80	2.53
Iron	22.60	28.00	49.90	43.70	31.70	30.40
Lithium	28.80	27.70	37.50	32.00	24.20	22.70
Magnesium	3.97	3.20	4.78	3.08	3.48	3.44
Manganese	1.27	-	1.19	-	-	0.91
Sodium	36.80	45.00	61.40	41.30	68.90	59.60
Nickel	16.70	17.10	18.60	16.10	14.00	19.30
Silicon	-	-	4.30	4.70	7.73	7.60
Titanium	3.00	3.60	3.93	3.91	3.20	2.50
Zinc	1.08	1.10	1.29	1.23	1.71	1.80





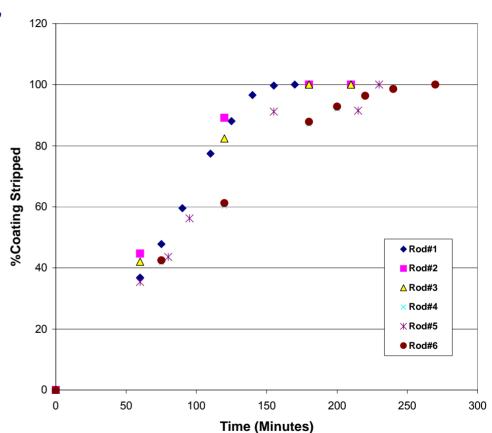
Stripping Solution Development

Citric / Peroxide Solution

 Effective electrolytic stripping solution, but unstable

Alkaline Dip Stripping Solution

- Initial studies performed on 1"x4"
 Samples with ~0.004" nCoP coating were stripped (dip)
- Removal rate averaged ~0.001" per hour from fresh solutions
- Solution was found to be stable with use
- Rate of dissolution was found to decrease with use.
- No attack of steel substrate has been observed, even with used/aged solutions.
- Solution has been further validated:
 used to strip 0.002" thick nCoP
 coating from six (6) grinding rods
 (approx 12g coating penrod) or particles of the coating penrod) or particles of the coating penrod or particles of the c







Technology Transfer to NADEP-JAX

Installation in old Chrome Plating Line

- 350 gallon old chromium tank has been prepared
- Bus bars installed
- Rectifier cables connected
- Partial delivery of chemicals has been received, with the remainder expected within the next two weeks
- Pulsed power supply has been installed in rectifier room and cables are in place









Tank Close-up and Bus-bar connections





- New steam heating being installed
- Solution agitation from filtration pump equipped with eductors
- Tank will be filled with distilled water and heated for a given period. Water will then be analyzed for any Cr6+ or other contaminants that may have leached out. If levels are too high, a drop in liner may be installed.





Pulse Rectifier Installation





Pulse power supply installed in rectifier room and water cooling lines were connected Technology Partnerships Canada



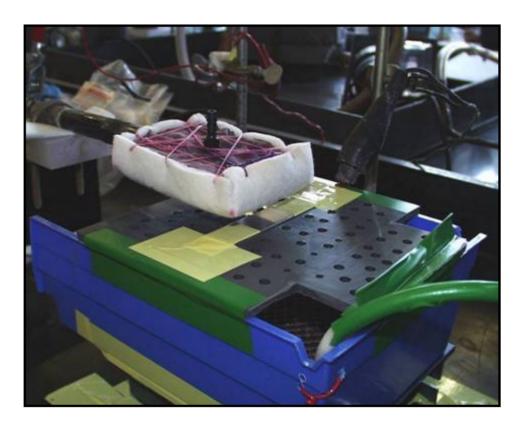
Selective Area (Brush) Plating Development



Selective Area (Brush) Plating Development

Overview

- Method to electrodeposit metal without tanks
- Well characterized industrial process
- Moving anode static cathode
- Can be easily field-implemented
- Can be applied manually, semiautomatic and automatic



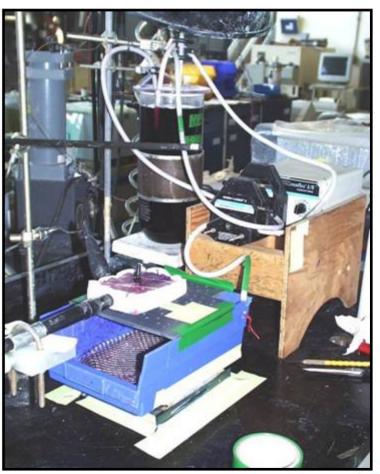


Selective Area (Brush) Plating Development

NTEGRAN

Set-up

- 3 L Plating Volume
- Adjustable flow rates using peristaltic and mag/drive pumps
- 6" x 6" perforated graphite anode
- Automatic anode oscillator with adjustable speed









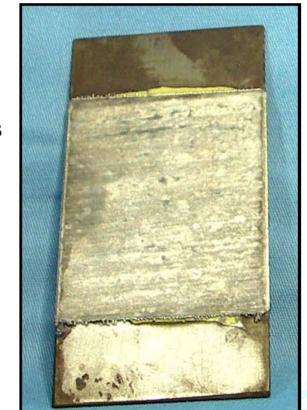
Brush Plating Procedure to deposit nCoP has been established

Process deposits at a rate of 0.006" per hour with a hardness of ~670VHN (measured at Integran) with a uniform composition through thickness

Adhesion on mild steel passes ASTM B571

Hardness independently measured at 734 VHN. Coefficient of Friction, per ASTM D1894-01, was independently measured to be 0.39 (Static) and 0.30 (Kinetic).

Process to be transferred to NADEP-JAX





Joint Test Protocol (JTP) Sample Prep and Testing







Summary Joint Test Protocol (JTP)*

- Demonstrate adhesion on various substrates
 - 4340, 15-5PH, Aermet 100, Al 7075, IN718 and Ti 10-2-3)
- Pre-Grinding Study
- Performance Testing
 - Axial Fatigue (ASTM E466-96)
 - Hydrogen Embrittlement (ASTM F519)
 - Corrosion (ASTM B117)
 - Fluid Immersion
 - Recipricating Sliding Wear
- Friction and Wear (Bushing) Tests

*Copy of JTP can be found on the HCAT website







Objective of grinding study is to determine a set of reasonable (but not necessarily optimum grinding conditions

Piston Rods (1" diameter) were coated with ~0.010" of nCoP for grinding studies

- 1st Batch of Rods coated and shipped to NADEP JAX (June 2005)
 - 4 x 4130 (2 As-deposited / 2 HT @ 191°C for 24hrs)
 - 2 x 15-5PH (As-deposited)
- 2nd Batch of Rods coated and shipped to NADEP JAX (Nov 2005)
 - 9 x 4340 Rods to be coated with 0.010" nCoP
 - 3 x As-deposited
 - 3 x HT at 191°C for 24hrs
 - 3 x HT at 300°C for 1-3hrs
 - 6 rods ground to ~0.002" thickness and returned to Integran for stripping
 - Rod stripped using alkaline chemical dip solution and returned to JAX (Dec 2005)





Piston Rods (1" diameter) coated with ~0.010" of nCoP were ground to a final thickness of ~0.002" (by Jon Devereaux at NADEP-JAX)

- Rods were ground following Mil-Std-866 using standard Al2O3 abrasives with no problems, ground very similar to chrome.
- Rod was easily finished to a 2-3μin roughness
- Stripped parts were examined after Nital etching and no evidence of any grinding burns was present







nCoP Coated 300M Pins

- 7 Rods Coated with 6 to 10mils nCoP
- Plated with 0.006"-0.010" of nCoP and ground down to 0.004" with Ra of 8µin.









Bushing Wear Testing

Rotational Friction and Wear Tests

- Tested to be performed at General Atomics coordinated by Paul Trester
- Rotational wear testing of nCoP coated 300M pins in contact with Ni-Al Bronze (AMS 4590, 130Ksi UTS Minimum)
- Testing will be performed at bearing pressures of: 10, 20 and 45Ksi
- Rotational tests will consist of +20° to 0° to –20° cycles at a frequency of 0.17Hz
- Diametral difference of ~0.001" between pin and bushing is desired to minimize grease loss during testing
- Surface finish of Ra=8µin is required in order to compare to previous test results of HVOF and Hard Cr.
- All the coated pins have been measured for diameter and surface finish (by GA) in the zone where the 0.5" wide bushing will contact the coated pin, at approximately mid-length of the coated zone.
- Friction and Wear Tests Scheduled at the GA-SI facility in Utah at within the next two months



Thin Dense Chrome Development



Thin Dense Chrome Alternative Development

Process and Properties of nCoP showed promise as potential alternative for TDC coating application.

With support from the JSF office, the process was developed to deposit coatings with four different phosphorus concentrations, 2-3, 4-5, 8-9 and 11-12wt%P (target coating thickness: 12µm (0.0005") and were tested for:

- Thickness Uniformity
- Surface Finish
- Morphology
- Adhesion (ASTM B571)
- Ductility (ASTM B489)
- Corrosion (Salt Spray (ASTM B117))
- Hardness
- Sliding Wear Testing
- Taber Abrasive Testing

Benchmark comparison made against TDC (AMS 2438A)

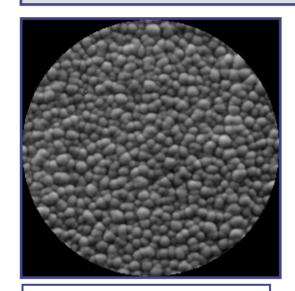


TDC Results

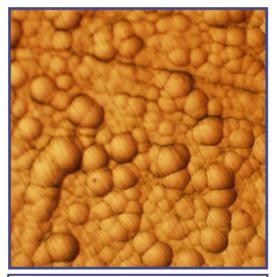


Surface Morphology and Hardness

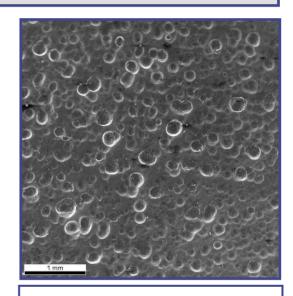
Surface morphology is **nodular** (similar to that of Thin Dense Chrome)



Thin Dense Chrome
Thickness ~0.0005"
Hardness
>900VHN



Co 2-3wt% P
Thickness ~0.002"
Hardness
~600-650VHN As-Dep



Co 11wt% P
Thickness ~0.002"
Hardness
~650VHN As-Dep







Property	2-3wt%P	4-5wt%P	8-9wt%P	11-12wt%P	
Thickness Uniformity	Need proper masking/shielding to achieve required thickness				
Surface Finish	Surface roughness unaltered after coating to 0.0005"				
Morphology at 0.0005"	Slightly Nodular		Smooth		
Adhesion	Pass	Pass	Pass	Pass	
Ductility	4-5%	~1%	~1%	~1%	
Corrosion Salt Spray (As-Dep) (HT@>250°C)	Pass Fail	Pass Fail	Fail Fail	Fail Fail	
Hardness (As-Dep) (HT)	600 VHN 750 VHN	650 VHN 850 VHN	650 VHN 1000 VHN	650 VHN 1100 VHN	
Wear (Sliding)	Good	Good	Good	Good	
Wear Abrasive (As-Dep) (HT)	TWI=18 TWI=22	TWI=22 TWI=25	TWI=32 TWI=10	TWI=29 TWI=7.7	

TDC Summary



Two Classes of Coatings Recommended

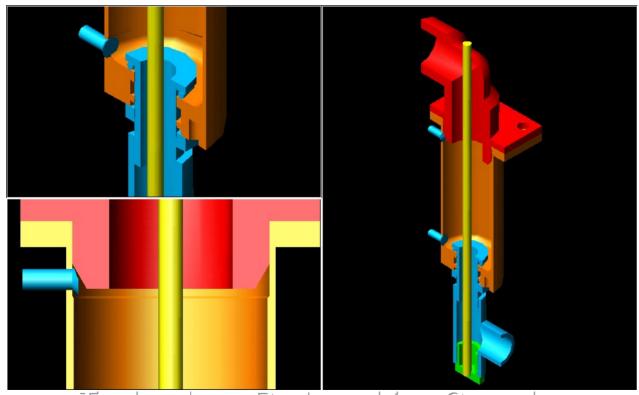
	Class 1 nCoP 2-3wt%P HT 191°C – 24hrs	Class 2 nCoP 11-12wt%P HT 300°C – 6hrs
Application Types	When corrosion resistance is required and the substrates cannot be HT to 250°C	When corrosion resistance is not required and the substrates can be HT above 250°C
Hardness	650 VHN	1100 VHN
Corrosion (ASTM B117)	Pass	Fail
Wear (Taber)	Good ~18mg/1000cycles	Very Good ~7.7mg/1000cycles

ESTCP

TDC Development



A Class 1a nCoP 0.0005" thick will be applied to a to two 3BSM actuator cylinders supplied by Smiths Aero for seal and wear testing coordinated by Malcolm Diplock. The anode/cathode supports for the plating jig are currently being constructed



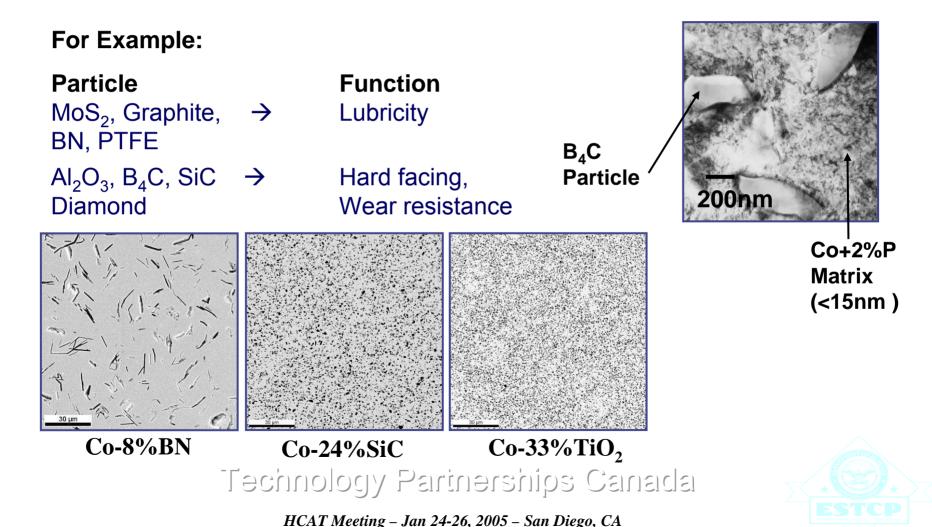






Nano Structured Composite Coatings

Further property enhancement can be achieved through the co-deposition of various **particulate** into the nanocrystalline matrix



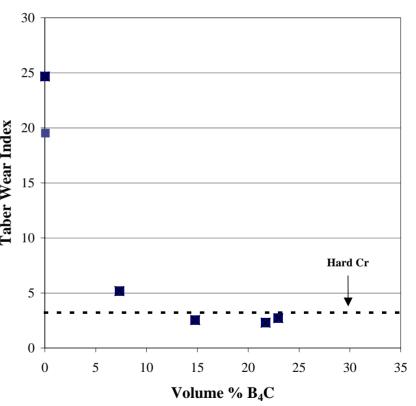


Nano Structured Composite Coatings

Nano CoP-B₄C SEM Micrograph



Abrasive (Taber) Wear



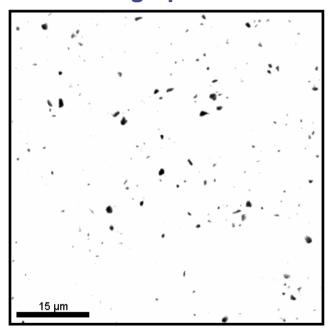
~22vol% B₄C





Nano Structured Composite Coatings

Nano CoP-Diamond SEM Micrograph



~3vol%Diamond

Taber Wear Index: ~1mg/1000cycles Surface Roughness: Ra=3μin at 12μm

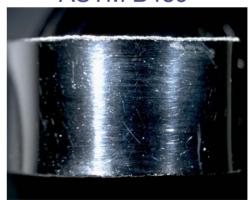
and Ra=3μin at 50μm

ASTM B117 - 48Hrs Exposure

0.0005" thickness: No red rust



Ductility: 4-5%
ASTM B489







Collaborative Projects





Goodrich Corp. Collaborative Project

As part of an industry effort to eliminate and replace chrome plating, the landing gear division at Goodrich is currently seeking a Cr-coating replacement in the ID surfaces of landing gears

Objective

Determine the suitability of nanocrystalline Co-P as an alternative to hard Cr coatings (4 \pm 1 mils thick) for NLOS applications

Method

Test samples provided by Goodrich

- Various high strength steel coupons (AISI 4130, 4340, 300M) as well as actual actuator cylinder (dummy part)

Processed at Integran Technology Inc.

- Preparation, pre- and post-plating procedures similar to those of the Goodrich process
- Plating of nano-CoP coatings (4 ± 1 mils in thickness)

Performance validation tests carried out at Goodrich Corp.

-Porosity, adhesion, hardness, hydrogen embrittlement, and salt-spray corrosion resistance

Technology Partnerships Canada





Porosity, Adhesion, Hardness Test Results

• Sample requirements for porosity, adhesion and hardness tests

Basis Material	Dimension ($w \times I \times t$)	Coating Thickness	Quantity
4130 steel annealed	1" × 4" × 0.040"	4±1 mils (100 ±25 μm)	18

Porosity Test

- A sheet of filter paper soaked with a ferroxyl solution at ~85 °C was placed and spread to the flat coated surface of the specimen.
- After 10 min, the heated filter paper was removed and examined.
- No dark blue spots resulting from corrosion of base metal interacting with the ferroxyl solution
- At a macro-level, nano-CoP plating produces a fully dense coating free from microcracks, pores or other defects that may reach the substrate.



Porosity, Adhesion, Hardness Test Results

Adhesion Test

Two separate activation procedures investigated:

- 1. Anodic etching in H₂SO₄-HF acid bath (350 mA/cm² for 1 min.)
- 2. Immersion in HCl solution (3 ~ 4 min.)

Adhesion testing consists of

- 1. Bend test (ASTM B517)
- No nano-CoP coating delamination occurred after bending the sample to fracture.
- 2. Flap peen adhesion test (BAC5730-2)
- Did not expose the base material after rotating the flap peen at 5200 rpm (min.) until 600% coverage was achieved

In all cases, nano-CoP coatings demonstrated "good" adhesion on the high strength steels



Porosity, Adhesion, Hardness Test Results

Hardness Test (ASTM B578)

 Microhardness values reported from three separate Co-P coated specimens (measured on the cross-sections using 100 g load)

	Nano-coating 1	Nano-coating 2	Nano-coating 3
Vicker's	695 ± 25.7	708 ± 64.3	696 ± 37.6

- The Vicker's hardness values were found to be \sim 700 H_{v}
- Exceeds Boeing Process Specification for normal hard Cr (BAC5709)
 requirement (H_v>595)





IV. Hydrogen Embrittlement (ASTM F519) Test

• Specimen requirements for the H₂ embrittlement test

Basis Material	Dimension ($w \times I \times t$)	Coating Thickness	Quantity
	ASTM F-519 Type 1a notched bar specimen	4±1 mils	
AISI 4340 steel		1 mm	16 (4 per batch)

Summary of test results

Batch	Activation	Post-plating Condition	Pass/Fail
1	H ₂ SO ₄ -HF anodic	12 hours at 191°C	Pass
2	H ₂ SO ₄ -HF anodic	No hydrogen bake	Fail
3	Immersion in HCI	No hydrogen bake	Fail
4	No Activation	No hydrogen bake	Fail

Hydrogen bake-out treatment is necessary to eliminate any embrittling characteristics of plating on high strength steels.

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VI. Salt-spray Corrosion Test (ASTM B117)

• A list of samples for the salt-spray corrosion test

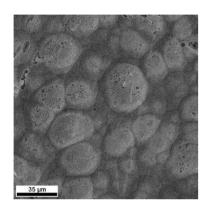
Coating Material	Basis Material	Dimension $(w \times l \times t)$	Coating Thickness	Quantity
Nano-CoP	AISI 4130 steel	4" × 6"× 1"	4±1 mils	1
Hard Cr (for comparison)	AISI 4130 steel	4" × 6"× 1"	4±1 mils	1

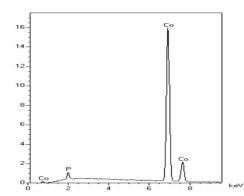




V. Salt-spray Corrosion Test (ASTM B117)

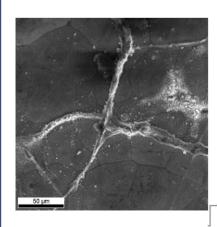
1. Nano-CoP coating after 200 hours

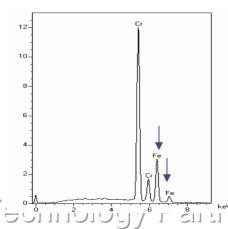




- No red rust spot formed after 200 hour exposure
- Uniform corrosion without showing any iron (substrate) corrosion product on the surface as confirmed by SEM/EDS examination

2. Hard Cr coating after 48 hours





- ~30% of the hard Cr coating surface was covered with red rusts, indicating severe corrosion of the substrate
- Preferential corrosion along the pre-existing microcracks as confirmed by SEM/EDS



ID Plating of Actuator Cylinder

- The ID surface (\sim 5" in diameter x \sim 12" in length) of actuator cylinder (dummy piece) was plated with nano-CoP coating (at Integran) in order to demonstrate Integran's process for NLOS application
- The cylinder was cut into five sections and evaluated at Goodrich
 - 1. Thickness variation through the cylinder was acceptable (as the measured thicknesses were between 0.0057" and 0.0067").
 - 2. Salt spray testing on both as-plated and lightly honed surfaces showed no-red rust spot even after 392 hours.
 - 3. The measured hardness values were >600 H_{v}
 - 4. A flap peen adhesion test revealed that the adhesion was acceptable (no substrate exposed), however, coating delamination was observed on some areas (likely due to scale up issues with the activation solution).

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After the salt-spray test (392 hours)



After the flap peen adhesion test







Goodrich Performance Validation Test

- Porosity, adhesion, hardness and corrosion resistance were found to be acceptable
- The same heat treatment as hard Cr post plating treatment (191°C (375°F) for 12 hours) was necessary to avoid hydrogen embrittlement of the high strength steel substrate
- Goodrich has expressed concern on the lack of fatigue data for this coating. An extensive study on fatigue resistance of nano-CoP coatings is in progress





Messier-Dowty Collaboration Project

Messier-Dowty is currently seeking a suitable alternative to Thin Dense Chrome (TDC, ~0.0005") in their landing gear manufacturing for NLOS applications.

Project Objectives:

- Demonstrate Adhesion on base metal substrates
- Design and fabricate plating anodes for plating 1" ID cylinders
- Test with mock-up geometries
- Develop property capability and demonstrate industrialization

Specific Performance Test:

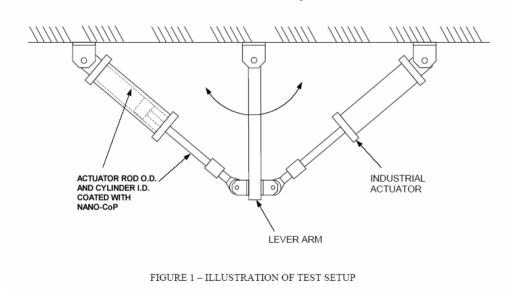
Assess the performance and durability of nCoP coatings in an utility actuator application for a cycle time of 20,000 cycles (one life) simulating a scheduled commercial overhaul inspection.







- 1. Nano-CoP coatings (0.0005" thick) were applied to
 - a. the OD surface of the actuator piston rods
 - b. the ID surface of the actuator cylinders

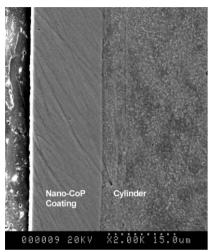


2. The nano-CoP coated industrial actuators were subjected to 20,000 cycles at a constant pressure of 3,000 psi throughout the duration of the endurance test. The inspection simulates a scheduled overhaul inspection.

Endurance Testing Results

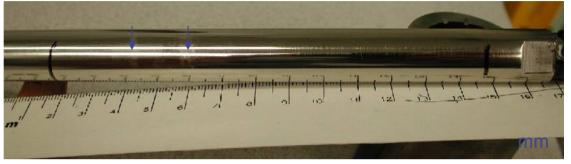


- As-plated Nano-CoP coating (0.0005" thick) was found to conform to substrate surface

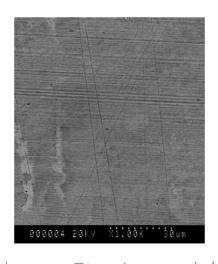


- Surface profilometer measurements showed that the roughness virtually stayed the same ($R_a = 0.06$) after plating
- 1 out of 3 cylinders showed poor adhesion as the coating delaminated during sectioning due to problems with activation procedure. Corrective action has taken place.

After the endurance test



- Minor wear/scoring (discoloration) was observed on the localized areas of piston and cylinder surfaces (e.g., as indicated by arrows)



- SEM examination of the wear bands revealed that the visually obtained wear is in fact, scoring on the order of sub-micrometer in depth (negligible wear)
- Acceptable leakage performance (<1 drop of hydraulic fluid in 25 cycles)

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Summary of Messier-Dowty Project

Messier Dowty Endurance Test

- -The technology demonstrator (landing gear utility actuator) was successful in performance goals.
- Based upon the results the coating is under consideration as a candidate for the A 380 nose landing gear design team as the NLOS coating.



SUMMARY



- Industrial scale-up of nCoP process successful to date
- Tank installation at NADEP-JAX underway and Technology transfer scheduled Feb 2006
- Brush Plating development completed
- JTP Test sample preparation is underway
- Two classes of nCoP coatings recommended for Thin Dense Chrome Type applications and will be further validated in component testing

